DEFORMATION AND ANNEALING RESPONSE OF TD-NICKEL CHROMIUM SHEET

by

R. D. Kane and L. J. Ebert

ANNUAL STATUS REPORT

July 1973

Prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Grant NGR 36-003-094

School of Engineering
Department of Metallurgy and Materials Science
Case Western Reserve University
Cleveland, Ohio 44106

PRICES SUBJECT TO CHANGE

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Submitted to:

Office of Grants and Research Grants
Attention Code SC
National Aeronautics and Space Administration
Washington, D. C. 20546

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I

DEFORMATION AND ANNEALING RESPONSE OF TD-NICKEL CHROMIUM SHEET

ABSTRACT

by

Russell D. Kane

The deformation and annealing response of TD-nickel chromium (TD-NiCr) 0.1 inch thick sheet was examined using various cold rolling and annealing treatments. Upon annealing (above 816°C (1500°F)), the as-received material was converted from an initially ultra-fine grain size (average grain dimension 0.5 - 1 micron) to a large grain structure. Increases in grain size by a factor of 100 to 200 were observed for this transformation. However, in those material states where the large grain transformation was absent, a fine grain recrystallized structure formed upon annealing (above 732°C (1350°F)). The deformation and annealing response of TD-NiCr sheet was evaluated with respect to the processing related variables as mode and severity of deformation and annealing temperature. Results indicate that the large grain transformation in TD-NiCr sheet is abnormal grain growth. In the absence of this transformation, classical primary recrystallization occurs.

Using selected materials produced during the deformation and annealing study, the elevated temperature tensile properties of TD-NiCr sheet were examined in the temperature range 593°C (1100°F) to 1093°C (2000°F). It was observed that the elevated temperature tensile properties of TD-NiCr sheet could be optimized by the stabilization of a large grain size in this material using the cold working and/or annealing treatments developed during the present investigation.

Limited studies were conducted by Department undergraduates in the areas of deformation and annealing response of TD-NiCr sheet. The starting materials

were (1) the 0.275 inch thick sheet and (2) the 0.1 inch thick sheet following annealing at 1500°F (816°C) for 90 hours. It was observed that grain size and L/D ratio of the 0.275 inch thick sheet were relatively insensitive to the processing-related variables as mode and severity of deformation and annealing temperature. It was possible to produce material states of TD-NiCr from the 0.1 inch sheet which were characterized by large grain sizes and L/D ratios of about 30 by using repeated combinations of cold rolling and annealing.

FOREWORD

This Annual Status Report represents investigations conducted under NASA Grant NGR 36-003-094 from June 1972 to June 1973. The initial portion of this presentation describes the work presented to Case Western Reserve University by Russell D. Kane in partial fulfillment of the requirements for the degree of Master of Science. Additional data obtained during the course of the past year by (unsupported) student projects has been included in the Appendix of this report.

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INTRODUCTION

In recent years, much has been learned of the recrystallization characteristics and of the elevated temperature deformation mechanisms of TD-Nickel [1-8], the forerunner of TD-Nickel-Chromium (TD-NiCr). However, the recentness of the development of TD-NiCr has not permitted ample opportunity to evaluate the material in light of the work which has been done on either the SAP (Sintered Aluminum Powder) or TD-Nickel alloys. Even more importantly, it was not known whether the unique recrystallization response[2,3] and the high temperature deformation mechanisms[4,5] which make TD-Nickel a very unusual alloy as also characteristic of TD-NiCr. Such knowledge becomes mandatory if optimization of high temperature use of TD-NiCr materials is to be achieved.

The primary purpose of the present study was the determination of the nature of the recrystallization and grain growth processes in TD-NiCr sheet material, and their evaluation in terms of classical primary recrystallization and abnormal grain growth concepts. Research performed on TD-Nickel has indicated that the development of an understanding of these phenomena played a key role in the development of processing procedures with which the elevated temperature properties of TD-Nickel could be brought to their full potential[4]. Because of the inherent similarities of TD-Nickel and TD-NiCr, it was felt that such an understanding would be equally important in gaining the optimization of the high temperature mechanical properties of the more recent TD-Nickel-Chromium material.

MATERIALS AND PROCEDURE

For the present study, TD-Nickel-Chromium (TD-NiCr) was supplied by the Fansteel Company in the form of 0.1 inch thick sheet. The product is fabricated by conventional powder metallurgical techniques. Thoria containing nickel-

chromium powder (approximately 80% nickel - 20% chromium) is consolidated and sintered, and then hot and warm rolled to final thickness. Typical composition and thoria content of this material is shown in Table I.

Deformation and Annealing

The deformation and annealing response of TD-NiCr sheet was determined with respect to the following parameters: mode and severity of deformation, and the time and temperature of annealing. In the present investigation the material was both longitudinally and transversely rolled without varying the rolling plane from that of the as-received material. These rolling operations were then followed by annealing treatments. The rolling was performed in step reductions of approximately 10% per pass. Annealing was carried out in an argon atmosphere for one hour at 1316°C unless specified otherwise.

To study the effects of the mode and severity of deformation on the final annealed grain structure in TD-NiCr, the unannealed, as-received TD-NiCr sheet was reduced 10%, 28% and 44% for both longitudinal and transverse cold rolling operations. The as-received and annealed material was rolled 12%, 21%, 28%, 48% and 67% in the transverse direction, and 53% longitudinally in separate operations. These materials were then annealed at 1316°C for one hour.

The influence of the annealing temperature in determining the final grain size was studied by annealing two different material states of TD-NiCr sheet at various temperatures between 732°C and 1316°C for two hours. The two material states chosen were the as-received material, and the as-received and annealed material reduced 48% by transverse rolling. As a corollary to this study, the effect of annealing time on the final annealed grain structure was determined by using the same material states and annealing temperature as those mentioned previously, but reducing the annealing time from two hours to one minute.

Grain Size and Microhardness Measurements

The grain size in the annealed materials was determined as a function of the severity and mode of prior deformation and annealing temperatures. In general, the grain shapes were not equiaxed; therefore, measurements of the grain dimensions were made in the rolling direction and in the transverse and thickness directions. The grain dimensions in each direction were determined by a line intercept technique and were the result of over 25 or more separate observations. By using a statistical analysis, the average grain dimension was found. This value was the statistical mean of over 200 separate grains.

To delineate the grains preparatory to grain size measurements, the various material states of TD-NiCr were metallographically polished and etched using either one of two etching techniques. The first consisted of electrochemical etching with a solution consisting of 6 parts ethanol, 1 part H_2SO_4 and 21 parts water using a 5-volt power. The second was a thermal etch and consisted of heating the polished specimen at 593°C for 5-10 minutes[9].

In addition to determining the grain size of the deformed and annealed materials, the microhardness of selected specimens was measured. The most important use of the microhardness readings was that associated with the characterization of transformed and non-transformed regions in the TD-NiCr sheet. A 300 gram load was employed in the microhardness measurements. Prior to testing, the specimen surface was either thermally or electrochemically etched by the techniques noted above.

Elevated Temperature Tensile Testing

In order to investigate the effect of grain size on the elevated temperature tensile properties of TD-NiCr sheet, tensile tests were performed on selected material states of TD-NiCr. These materials were produced by use of

the processing methods developed during the deformation and annealing study. Tests were conducted in the temperature range 593°C (0.52 T_m) to 1093°C (0.82 T_m) on (average) grain sizes of TD-NiCr sheet between 0.0037mm to 0.0515mm.

The elevated temperature tensile tests were performed in a vacuum of approximately 5×10^{-5} torr. The temperature was controlled to $\pm$ 2.8°C by means of a platinum - 10% rhodium thermocouple in contact with the middle of the gage section of the tensile specimen. The specimen design is shown in Figure 1. Generous radii of curvature at the specimen shoulders were employed to minimize shoulder breaks, thus insuring reliable measurements of ductility. For all specimens tested, the initial strain rate was 0.01/minute for the 0.5 inch gage section.

RESULTS

Deformation and Annealing

Upon cold rolling and subsequent annealing at 1316°C of the as-received, unannealed TD-NiCr 0.1 inch thick sheet, the average grain dimension (AGD) was found to decrease with increasing severity of prior deformation for both longitudinal and transverse rolling as shown in Figure 2. The true rolling strain has been used to indicate deformation to permit a more basic representation of the data. The combination of transverse rolling and annealing was observed to have a somewhat greater effect on reducing the AGD than did similar longitudinal rolling and annealing treatments.

Figure 3 shows similar curves for the rolling and annealing of TD-NiCr sheet where the starting material is in the as-received annealed condition. The AGD following these treatments was also found to decrease with increasing rolling severity for both longitudinal and transverse rolling. It was observed that the

decrease in grain size observed was due to a monotonic decrease in each of the three linear grain dimensions. It was also noted that the L/D* (grain aspect) ratio remained approximately the same during rolling and annealing. Values of L/D determined during this portion of the study were consistently between 2 and 4.

Five different material conditions of TD-NiCr sheet that were chosen as "standard states" for further investigation are shown in Table II. They have essentially a constant L/D ratio and differ only in grain size (grain volume).

The effect of the value of the annealing temperature on the grain size in TD-NiCr sheet is shown in Figure 4. The AGD of material state A (see Table II) following annealing was shown to increase with decreasing annealing temperature over the range 954°C to 1316°C for constant annealing time (2 hours). Only very small, insignificant effect was observed when material state B was transversely rolled 48% and annealed.

Figure 5 through 7 show the microstructure in material state A after annealing times of 1 minute to 2 hours at 926°C. After 2 hours at temperature, the entire specimen was transformed to a relatively large grain structure (AGD $\simeq$ 0.09mm). With shorter annealing times, the amount of material transformed to this structure is considerably less. As shown in Figures 8a and 8b, the untransformed areas appear as regions of extremely fine grains. The microhardness of these untransformed regions was not comparable to that of the initial material, but was, as shown in Table III, considerably less. It was only slightly higher than the regions which had undergone complete transformation to a large grain structure.

A similar investigation was performed on material state B following 48% transverse rolling. For annealing at 926°C, the material was completely transformed to a fine grain structure (~0.004mm) after only one minute at temperature

*Ratio of average length to average diameter of elongated grains.

with little observable grain growth for times up to 2 hours. However, at 732°C this material state is not transformed to a high degree until approximately nine minutes (as shown in Figures 9 and 10). Microhardness measurements show that there is little observable decrease in hardness until the material transforms to the fine grain structure, and then there is no further significant change once the transformation is complete.

Elevated Temperature Tensile Testing

Using material states B, L, C. and D, the standard tensile properties consisting of tensile strength, 0.2% offset yield strength and ductility (in terms of percent elongation) were measured over the temperature range 593°C to 1093°C. Figure 11 shows the tensile and yield strength of TD-NiCr sheet material as a function of temperature. All stress values were observed to decrease uniformly with increasing temperature. The grain size dependence of the tensile and yield strength is shown particularly well in Figure 12. Over the range of grain sizes tested, it was observed that there is a relatively large increase in the strength properties with grain size in the smaller grain size region (0.0037mm to 0.0067mm) but thereafter, these properties are nearly independent of grain size.

Figure 13 shows the ductility of these materials as a function of temperature for various grain sizes. At 593°C, all elongation values were clustered around 4 ± 2 percent. As the temperature was increased, the ductility began to show a definite grain size dependence. In the temperature range 982°C to 1093°C, the ductility increases significantly with decreasing grain size.

DISCUSSION

On the basis of the results of the present study it appears that two basically different annealing responses of TD-NiCr sheet were observed, depending on the initial state of the material and/or on the processing history prior to annealing. The as-received TD-NiCr 0.1 inch thick sheet (material state A) exhibited a particularly large increase in grain size upon annealing. The characteristics of this transformation are such as to indicate that abnormal grain growth processes were present. However, subsequent rolling and annealing produced a decrease in grain size with increasing rolling severity. This effect was very similar to classical primary recrystallization in pure non-thoriated metals.

Assessment in Terms of Abnormal Grain Growth

A complete discussion of abnormal grain growth in pure metals, as well as in dispersion strengthened metals, is given by Hillert[10] and is critically reviewed by Petrovic[3,11] in relation to TD-Nickel dispersion strengthened system. The importance of the particle dispersion in promoting abnormal grain growth arises from its effect in terms of the following equation for a normal grain growth in dispersion strengthened alloys:

$$V = M\gamma(1/R_{cr} - 1/R-Z);\ Z = 3kf/4r$$

where the boundary mobility, M, boundary surface energy, γ, actual grain radius, R, grain radius at V=0, R_{cr}, and grain growth rate, V, are related as shown. The term γZ, is the Zener drag term, k is a constant of value 1.5, f is the volume fraction of dispersoid phase and r is the dispersed particle radius. This equation predicts that normal grain growth should cease when $R_{cr} = 1/Z$ and that this is an inherent property of dispersion hardened materials. Therefore, abnormal grain growth develops from normal grain growth if two conditions are

simultaneously fulfilled:

(1) Normal grain growth ceases at an average matrix grain size below the value of 1/Z, and

(2) There must be at least one grain larger than average to act as a nucleus for abnormal grain growth.

For TD-NiCr, the value of 1/Z is approximately 1 micron where f = 0.02, r = 200Å and k = 1.5. As shown in Figure 8 the size of the fine grains produced prior to the large grain transformation were approximately 1.4 microns. Because of the approximate nature of the calculation of 1/Z, it is justified to conclude that recrystallization and normal grain growth has ceased near this value. It is also evident (see Figures 5 through 8) that there are isolated grains larger than this average size which do act as nuclei for the large grains transformation. These observations indicate that the conditions for abnormal grain growth have been fulfilled in this material state of TD-NiCr.

Further investigation also supports the presence of these processes. Hardness measurements taken at various times during annealing showed considerable softening occurring on the formation of the fine grains prior to the large grain transformation. This indicated the existence of recovery and/or recrystallization before the formation of the large grains. Observations were also made which indicate that the kinetics of the large grain transformation in TD-NiCr (at 926°C) are significantly slower than those processes occurring in material states of TD-NiCr where this transformation was absent, thus showing the sluggish nature of the large grain transformation. The grain size following annealing was found to decrease with increasing annealing temperature. All of these findings are consistent with those of Petrovic for abnormal grain growth in TD-Nickel.

Assessment in Terms of Primary Recrystallization

While the as-received TD-NiCr sheet fulfilled the conditions for, and exhibited the characteristics of, abnormal grain growth during annealing, that material rolled from the as-received and annealed TD-NiCr sheet (material state B) behaved quite differently upon annealing. This material exhibited a decrease in the final annealed grain size with increasing amounts of prior deformation. Such behavior is predicted by primary recrystallization. For a constant annealing temperature, the number of recrystallization nuclei increases with deformation severity as a result of the increased distortion of the lattice and the accompanying increase in stored strain energy. The increased number of nuclei in the material with increased deformation, results in a decrease in the grain size following annealing.

Using microhardness measurements and electron microscopy, it was found that little observable recovery occurs before the transformation to relatively perfect grains. Similar findings have been reported for primary recrystallization in pure nickel[12,13,14]. As shown in Figure 10, the average size of the recrystallized grains in these material states of TD-NiCr was approximately 4 to 6 microns.

In agreement with the argument presented in the previous section, it must be concluded that primary recrystallization and normal grain growth occurs in these material states because the conditions for abnormal grain growth were not satisfied. The experimental findings of the present study support this conclusion. The as-received TD-NiCr sheet formed a very fine grain structure comparable to the value 1/Z of about 1 micron before transforming to the large grain size, whereas the material rolled from material state B formed recrystallized grains of about 4 to 6 microns in size which stabilized at a size greater than 1/Z.

Therefore, the grain size in this material state was limited to that attainable by recrystallization and normal grain growth processes.

The present study also indicates that the particular annealing response may depend on the initial grain structure. The as-received material which exhibited abnormal grain growth characteristics upon annealing was initially observed to have grains between 1/4 to 1 micron in size. The material states which showed primary recrystallization characteristics had a large grain size prior to rolling and annealing. Similar results were reported by Petrovic[3,11] for material states of TD-Nickel produced by cold rolling of bar material. It is possible that the ultrafine grain structure prior to deformation and/or annealing could influence the annealing behavior because of the close proximity of deformation defects to the grain boundaries. These boundaries could act as vacancy sources and sinks for deformation defects, thereby facilitating large scale recovery and recrystallization without increasing the grain size to a value greater than 1/Z.

Elevated Temperature Tensile Testing

Previous sections of this study have indicated that the grain size in TD-NiCr sheet can be significantly, yet predicably, varied by various deformation and annealing treatments. Because of the anticipated use conditions for this material, the importance of grain size control lies in the effect of the microstructure in determining the elevated temperature load carrying capabilities.

Recent theories[4,5,8] have attempted to explain elevated temperature deformation of dispersion strengthened alloys in terms of two co-existing mechanisms: (1) grain boudary sliding and (2) grain interior deformation. It can be predicted that, as the grain size increases, the relative contribution of grain boundary mechanisms will decrease because of the decrease in the grain

boundary area. These mechanisms become increasingly significant with temperature (above 0.5 T_m). At temperatures above 0.5 T_m, the grain boundaries are weaker than the grains themselves. According to these theories, the elevated temperature strength properties will increase with increasing grain size, whereas the ductility will decrease. The results of the present study are in accord with these theories.

The elevated temperature mechanical properties of TD-NiCr sheet are also important in relation to those reported for TD-Nickel. The yield strengths of both are shown in Figure 14 (TD-Nickel data from Petrovic[4]) as a function of grain size. The only major difference in the materials is the addition of 20% chromium in solid solution in TD-NiCr. At 593°C (0.52 T_m) the higher yield strength of TD-NiCr may be due to solid solution hardening since the temperature is still low enough for dislocation glide to be the major deformation mechanism. As the temperature is raised, and diffusion processes become more significant, the solution hardening effects of the chromium would be expected to decrease. Therefore, the flow stress differential separating TD-NiCr and TD-Nickel would also decrease. Such is the case observed at 1093°C shown in Figure 14.

CONCLUSIONS

(1) Two basic annealing responses of TD-NiCr sheet were observed. They depended on the initial state and/or on the processing history prior to annealing.

(2) The characteristics of the large grain transformation in TD-NiCr sheet are significantly different from those of primary recrystallization, and consequently, cannot be rationalized in terms of these concepts.

(3) The characteristics of this transformation indicate that abnormal grain growth processes are present.

(4) In those material states of TD-NiCr which did not exhibit the large grain transformation upon annealing, primary recrystallization was observed.

(5) The elevated temperature tensile properties of TD-NiCr sheet could be significantly altered by controlling the grain size in this material, using cold working and/or annealing schedules developed during this investigation.

(6) Because of the congruity between the deformation and annealing response observed in the present study for TD-NiCr and that reported for TD-Nickel, it appears that other similar dispersion strengthened alloys may behave in much the same manner. Therefore, the elevated temperature mechanical properties of these materials may be optimized by methods similar to those developed for TD-NiCr and TD-Nickel.

TABLE I

Chemical Composition of TD-NiCr Sheet
Lot 3829

Element	Weight %
C	0.0174
S	0.006
Cr	19.0
ThO_2	2.11
Ni	Balance

TABLE II

Material States*

Material State Designation	Reduction in Thickness by Transverse Rolling	Average Grain Dimension	L/D Ratio
B	0	0.0515mm	2.2
L	21%	0.0195mm	2.4
C	28%	0.0067mm	2.6
D	48%	0.0037mm	2.3
A	(as-received) (unannealed)	less than or equal to 0.001mm	-

*For all states, the starting material (prior to cold rolling) was the as-received material annealed at 1316°C (2400°F) for 1 hour. Final grain sizes and shapes were established by a 1 hour anneal at 1316°C (2400°F) after cold rolling.

TABLE III

Microhardness Data for Various Specimen Conditions

Specimen Condition	Knoop Microhardness
As-Received 0.1 inch thick sheet	409
As-Recieved 0.1 inch thick sheet/ 9 minutes at 926°C (1700°F) (Non-Transformed Regions)	332
As-Received 0.1 inch thick sheet/ 9 minutes at 926°C (1700°F) (Transformed Regions)	309
As-Received 0.1 inch thick sheet/ 1 hour at 1316°C (2400°F) (Material State B)	290
Material State B/ transversely rolled 48%	614
Material State B/ transversely rolled 48%/ 4 minutes at 732°C (1350°F) (Non-Transformed Regions)	586
Material State B/ transversely rolled 48% / 9 minutes at 732°C (1350°F) (Transformed Regions)	371

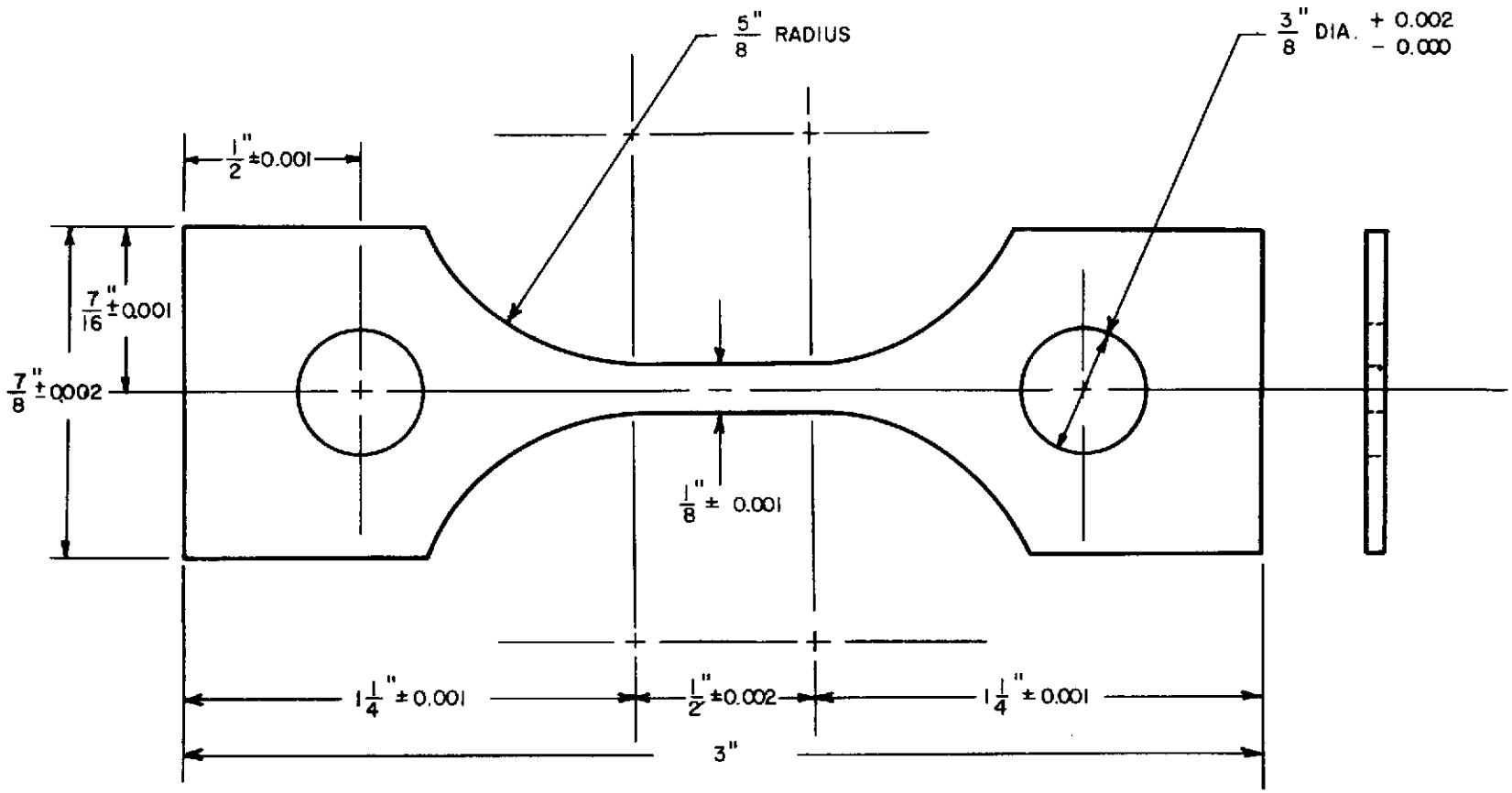


Figure 1. Sheet Specimen Geometry.

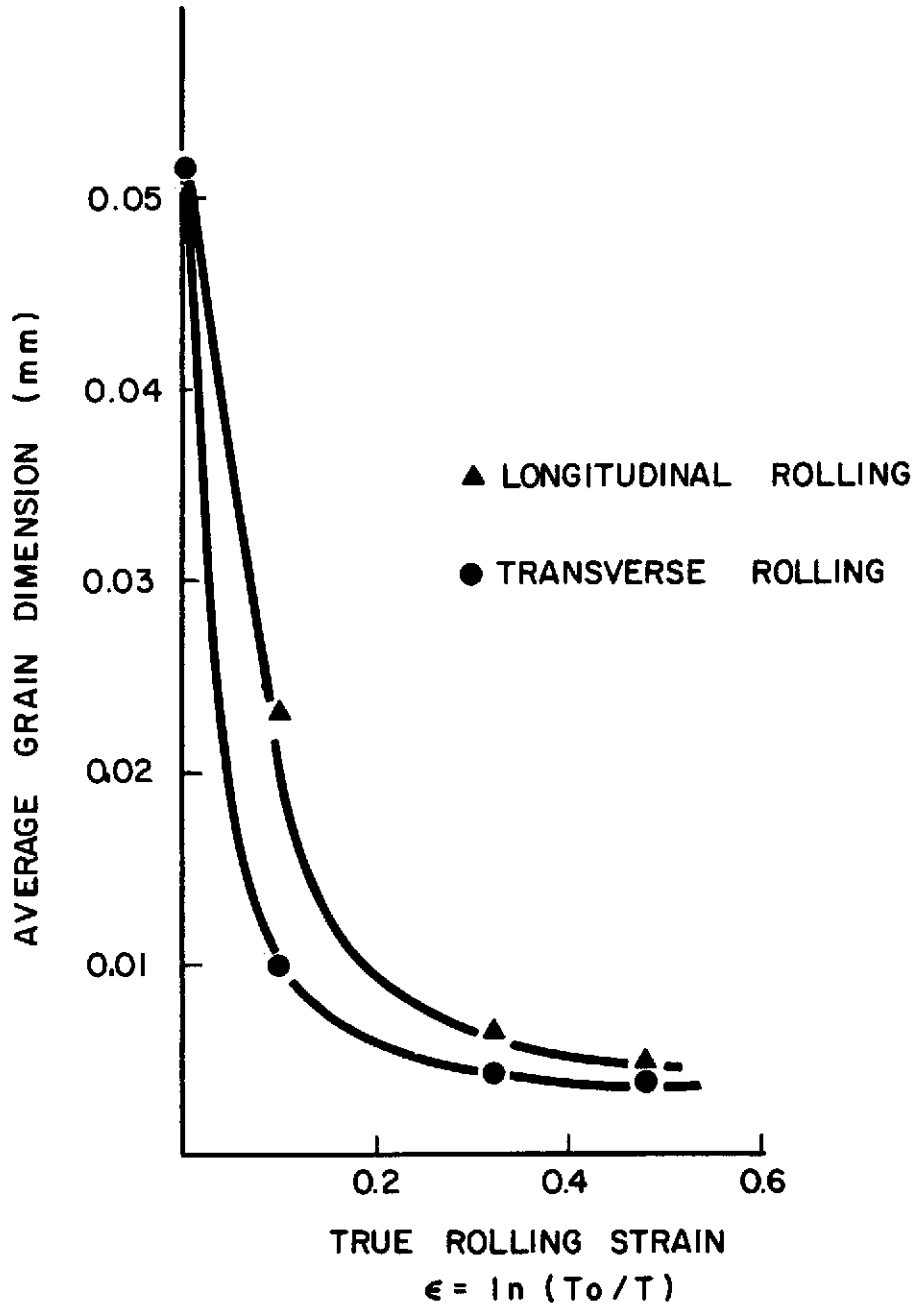


Figure 2. Effects of deformation severity on the average grain dimension for longitudinal and transverse rolling of the as-received material followed by annealing at 1316°C (2400°F) for 1 hour.

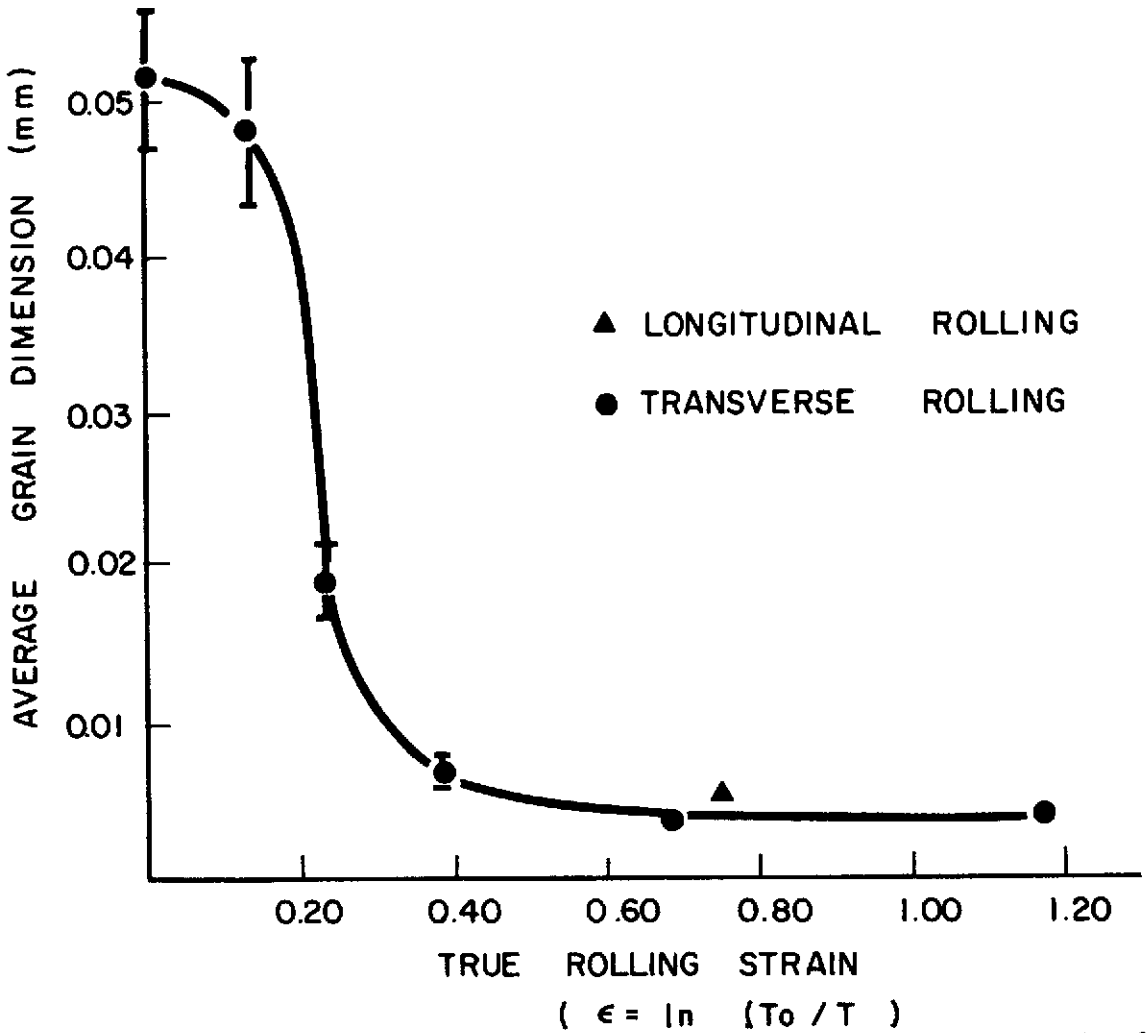


Figure 3. Effects of deformation severity on the average grain dimension for the rolling of material state B followed by annealing at 1316°C (2400°F), 1 hr.

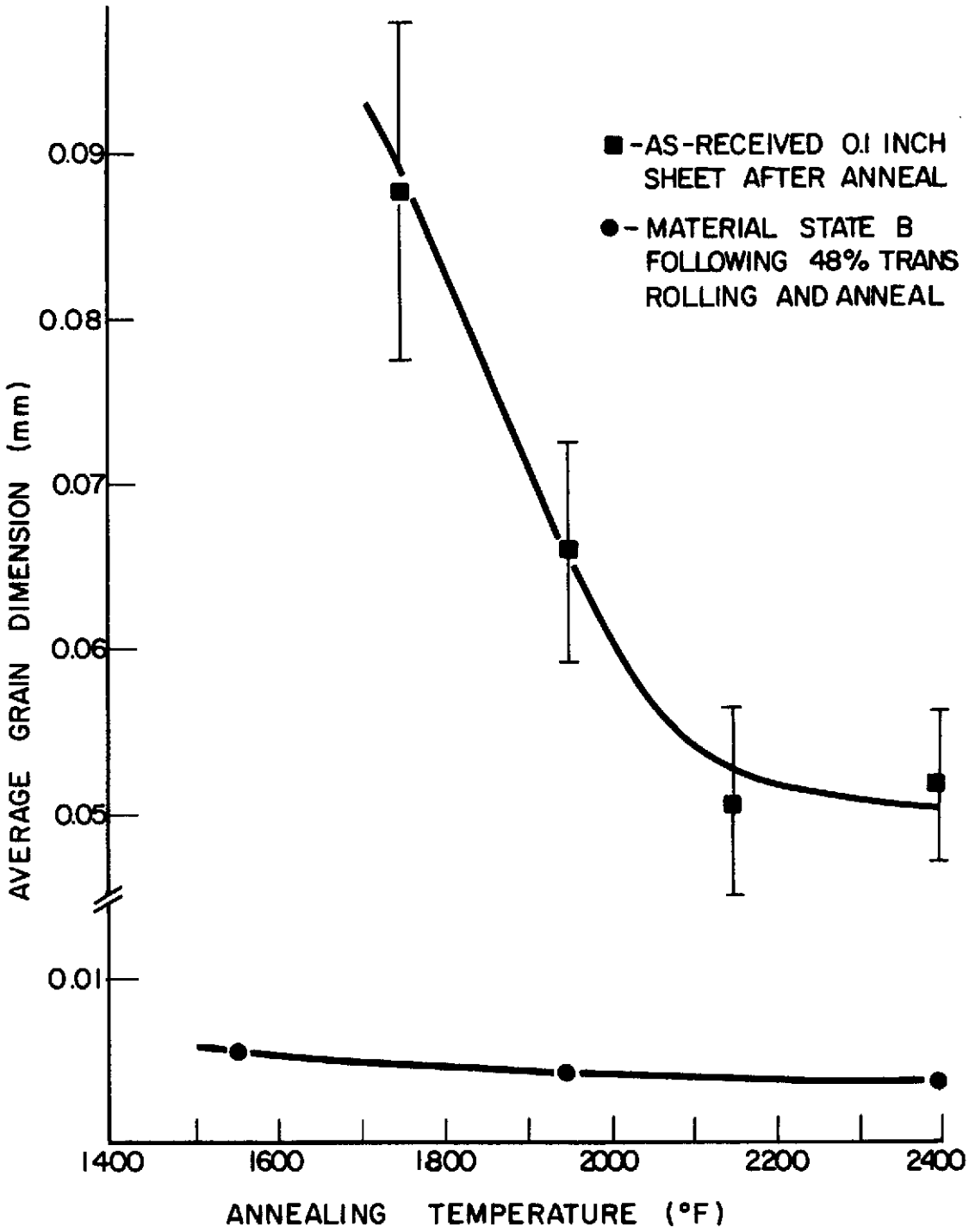


Figure 4. Effect of annealing temperature on the average grain dimension for annealing of a) the as-received material, b) material state B after 48% trans. rolling.

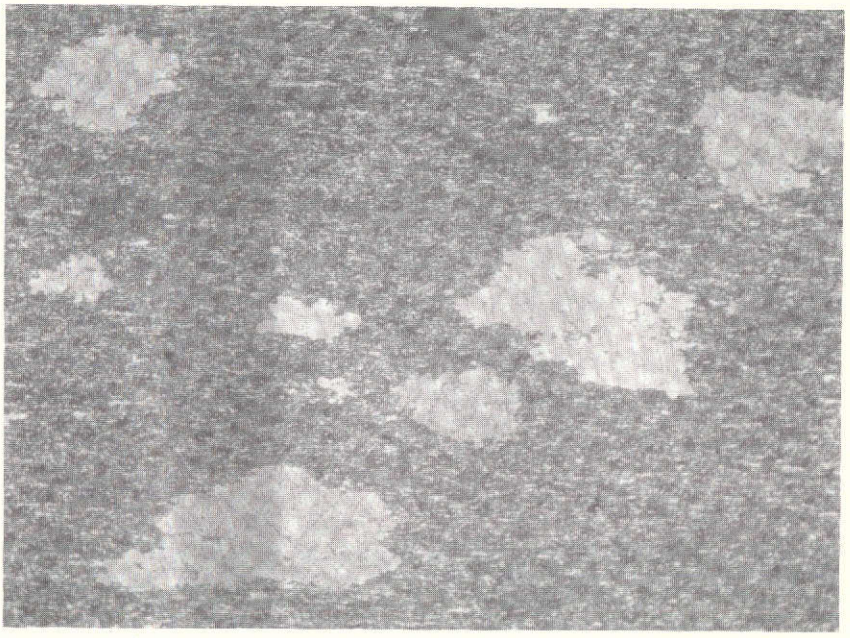


Figure 5. Microstructure of as-received material after annealing at 926°C (1700°F) for 4 minutes. Rolling plane - R.D. horizontal. (100X)

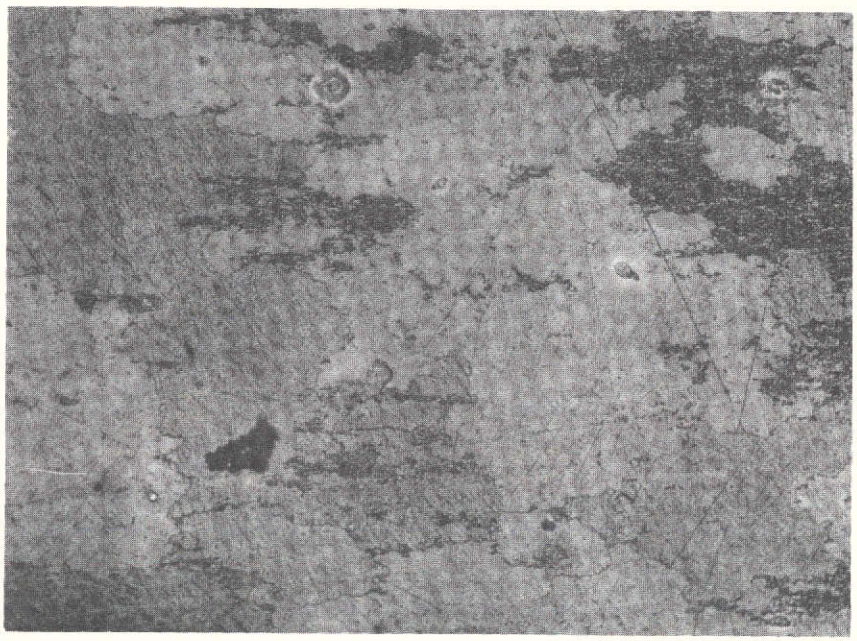


Figure 6. Microstructure of as-received material after annealing at 926°C (1700°F) for 9 minutes. Rolling plane - R.D. horizontal. (100X)

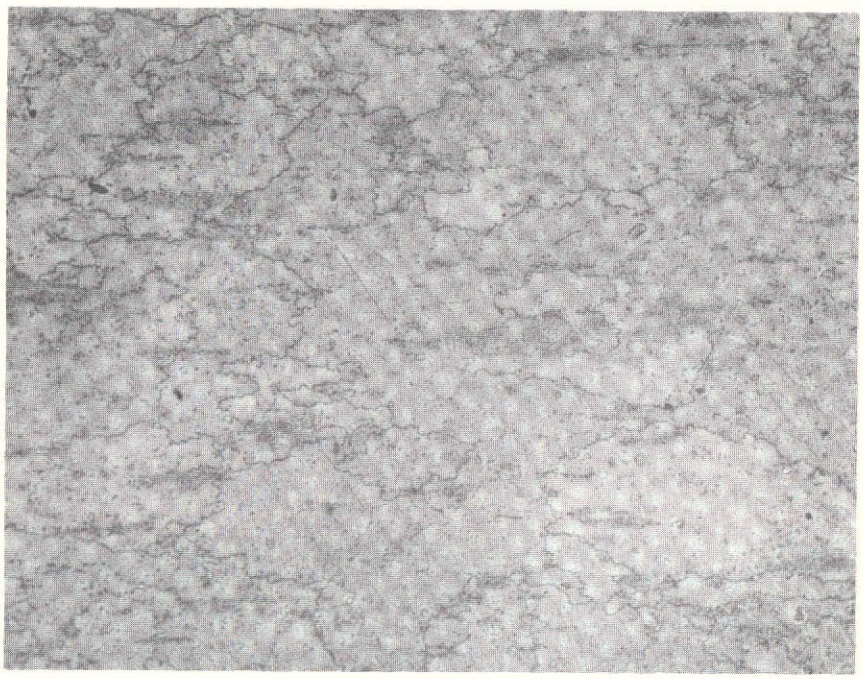
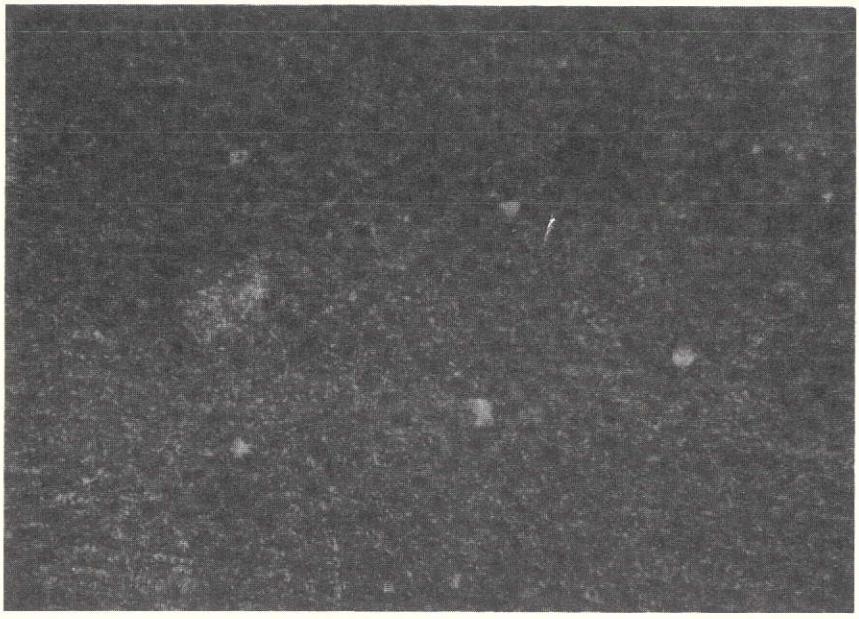
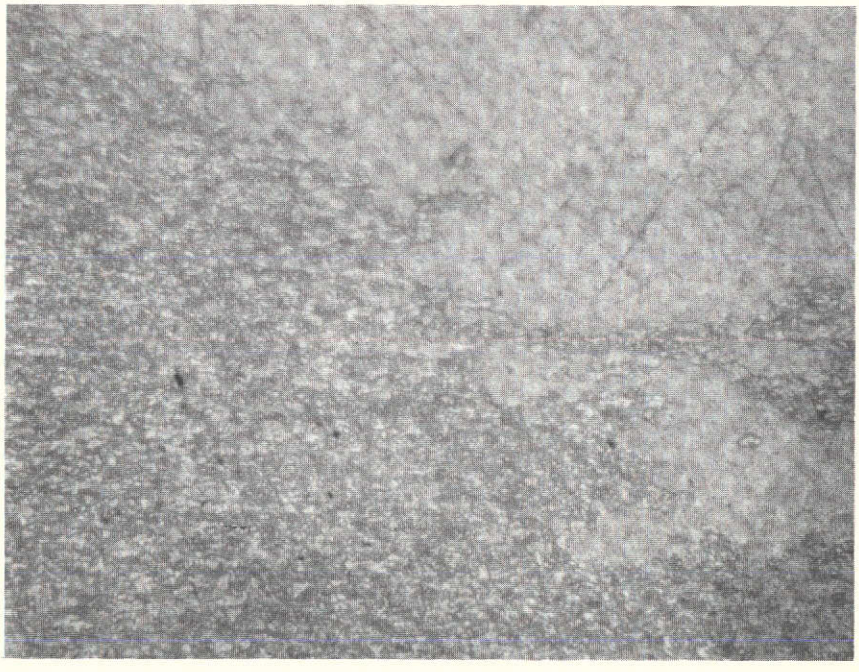


Figure 7. Microstructure of as-received material after annealing at 926°C (1700°F) for 2 hours. Rolling plane - R.D. horizontal. (100X)



a



b

Figure 8. Microstructure of as-received material after annealing at 926°C (1700°F) for a) 1 minute and b) 9 minutes. Rolling plane - R.D. horizontal. (500X)

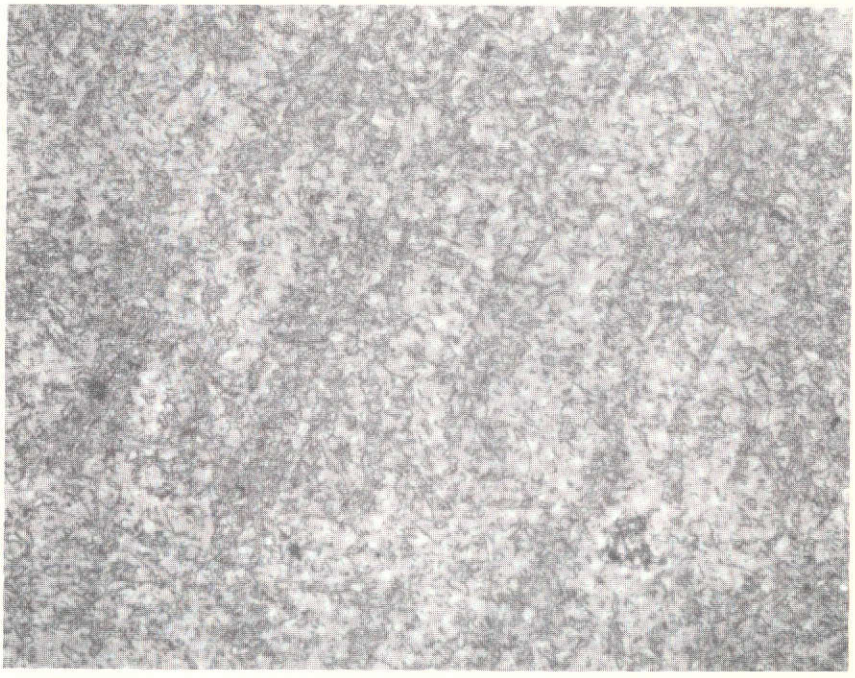


Figure 9 . Microstructure of material state B after 48% transverse rolling and annealing at 732°C (1350°F) for 4 minutes. Rolling plane - R.D. horizontal (300X).

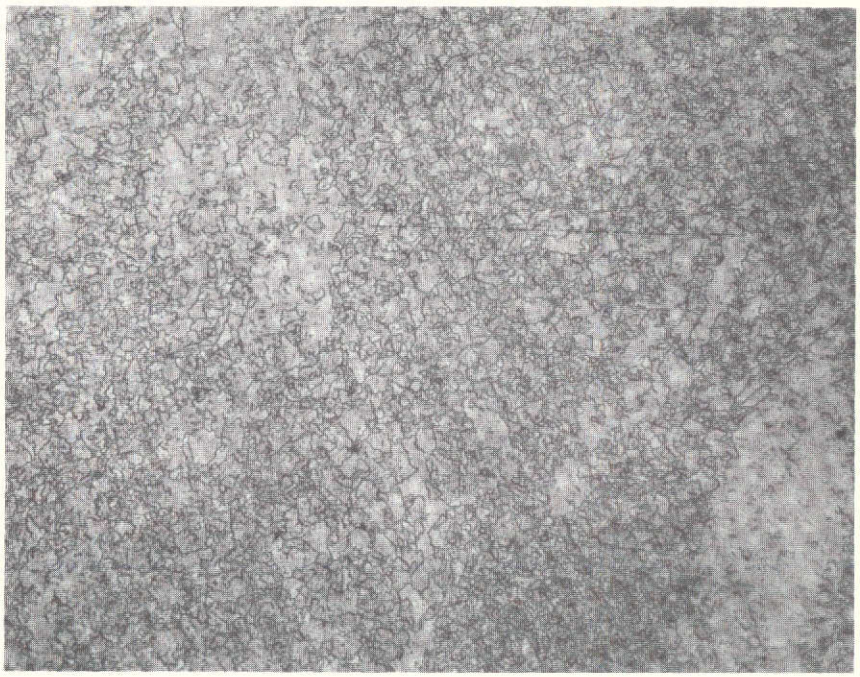


Figure 10. Microstructure of material state B after 48% transverse rolling and annealing at 732°C (1350°F) for 9 minutes. Rolling plane - R.D. horizontal (300X).

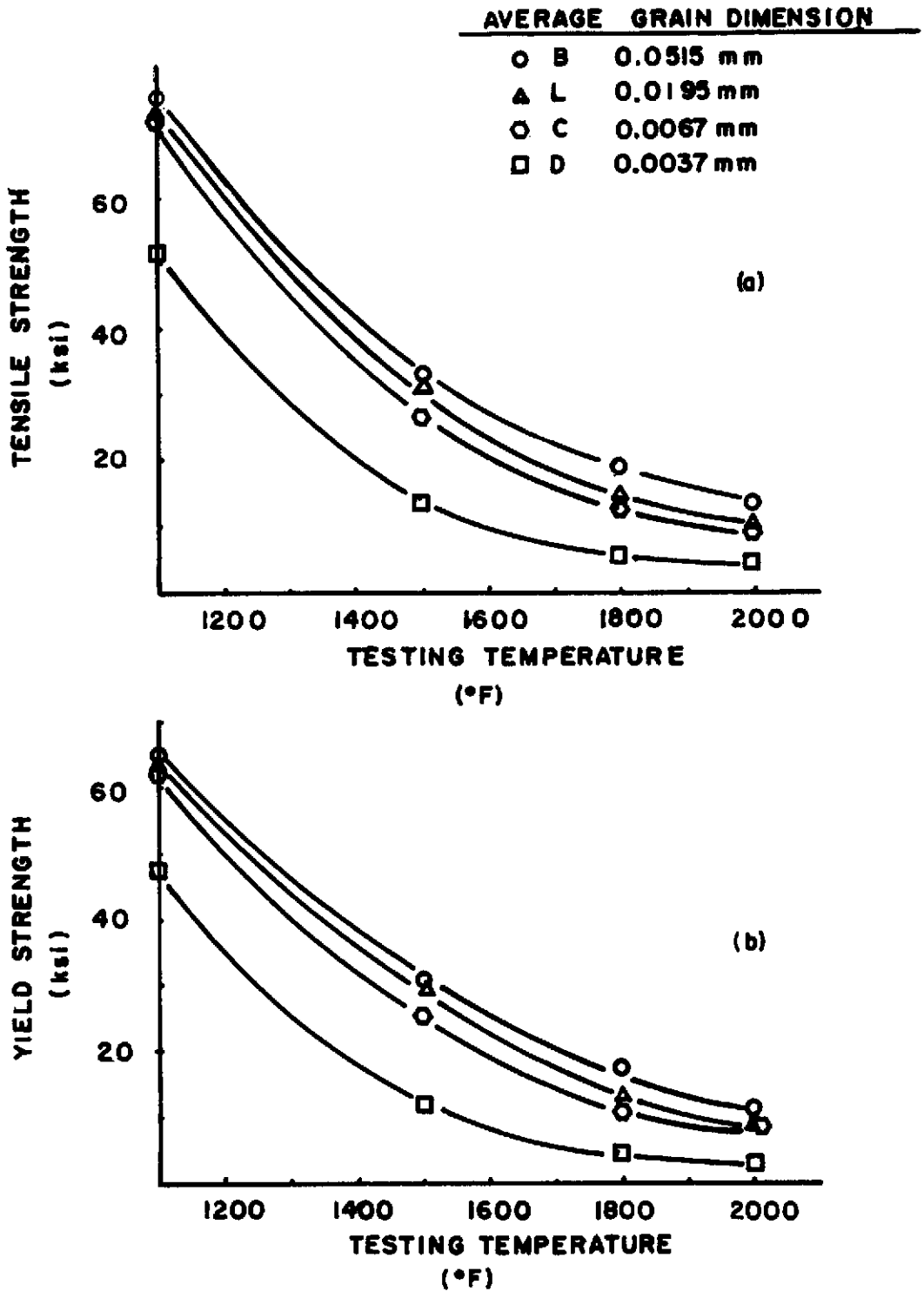


Figure 11. Effect of testing temperature on the elevated temperature a) tensile strength and b) yield strength for various material states of TD-NiCr.

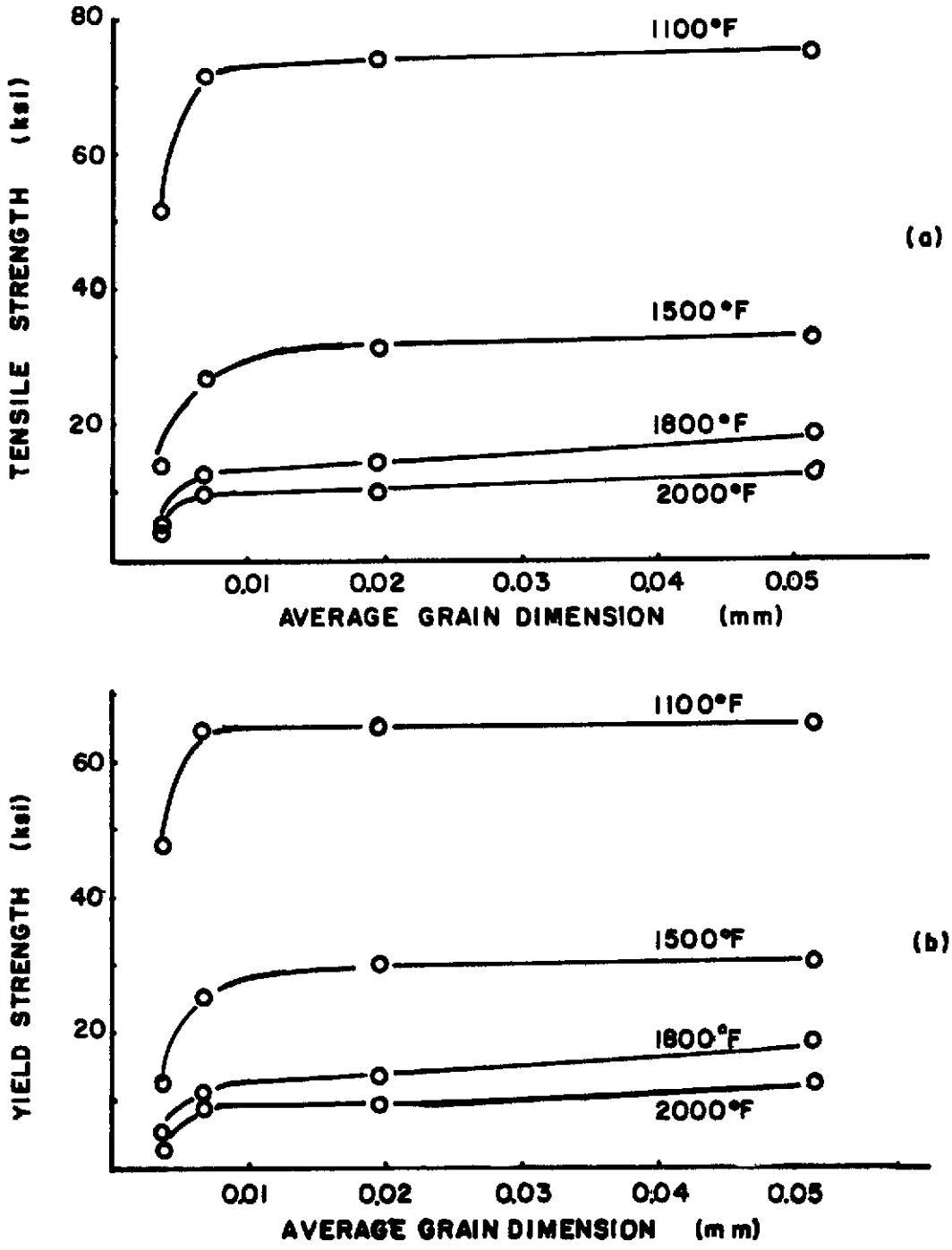


Figure 12. Effect of average grain dimension on the elevated temperature a) tensile strength and b) yield strength for TD-NiCr at various temperatures.

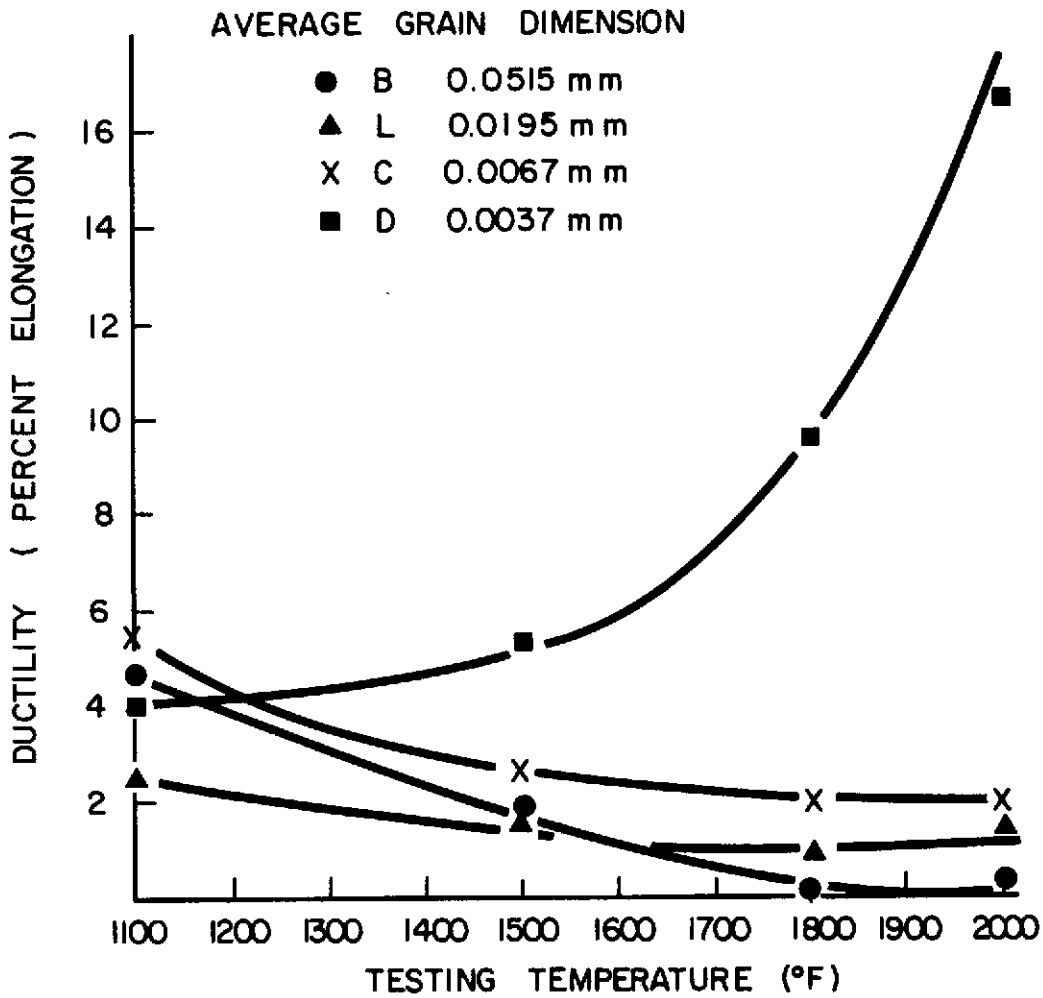


Figure 13. Effect of testing temperature on the ductility of various material states of TD-NiCr.

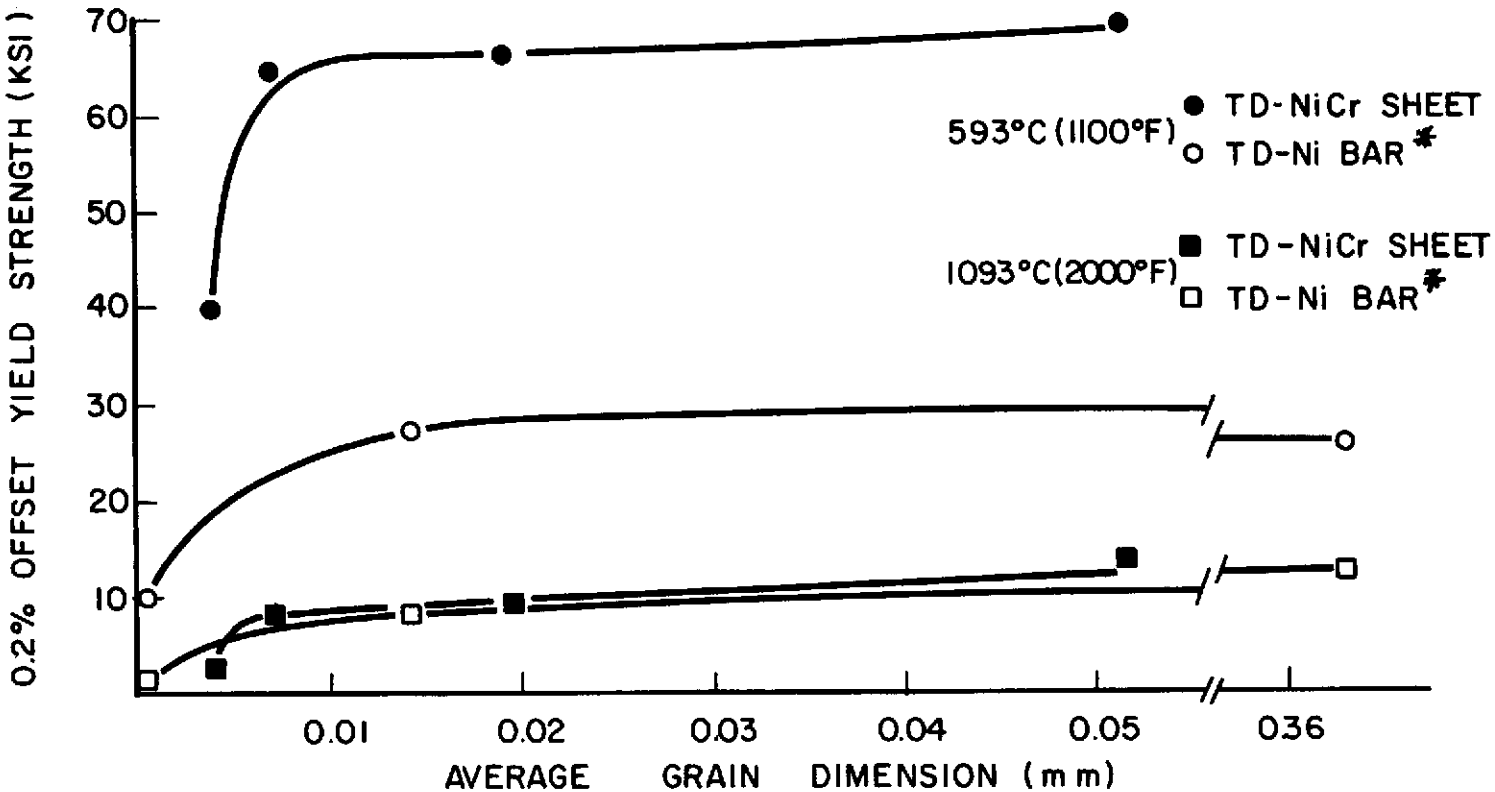


Figure 14. Comparison of the yield strengths of TD-NiCr sheet and TD-Nickel bar as a function of average grain dimension at 593°C (1100°F) and 1093°C (2000°F). *Data from reference 3.

APPENDIX

The results presented in this section were obtained during the course of the past year by (unsupported) student projects using the basic materials of this study. They are not necessary for support of the conclusions made in the preceding section. However, this data has been included for completeness because it does provide added information regarding the recrystallization and grain growth processes in TD-NiCr sheet materials.

This presentation has been separated into two parts, representing two different starting states of TD-NiCr sheet which were examined: (1) 0.275 inch thick TD-NiCr sheet (as-received condition), and (2) 0.1 inch thick TD-NiCr sheet (as-received condition plus annealing at 1500°F (816°C) for 90 hours). This latter material state will be designated B_{1500} in further discussions.

TD-NiCr 0.275 Inch Thick Sheet

A limited investigation of the deformation and annealing response of TD-NiCr 0.275 inch thick sheet was conducted. A portion of this work was in fulfillment of the Senior Project requirement (Spring 1973) of R.R.Hornack, an undergraduate in the Department. The purpose of this work was one of examining the effects of cold rolling severity and annealing temperature on the final grain size and shape in this material state of TD-NiCr sheet.

The as-received, 0.275 inch thick TD-NiCr sheet was separated into two lots, one annealed for 2 hours at 1500°F and the other annealed for 2 hours at 2000°F. Both lots were rolled longitudinally with respect to the original rolling direction. Rolling reductions of 20, 40 and 60% were made in steps of approximately 10% per pass. Following cold rolling, both lots of material were annealed in the same manner as before. Figure A-1 shows the average grain dimension and

grain aspect (L/D ratio) as a function of rolling severity. As indicated by Figure A-1a, the annealed grain size of this material decreases only slightly with increasing amounts of prior cold rolling. Initially, the TD-NiCr sheet had an annealed grain size of between 0.005mm and 0.007mm (5-7 microns); this decreased to only 3 to 4 microns after 60% reduction and annealing. In Figure A-1b it can be seen that L/D. ratio varies only between 1 and 2. It is evident from these plots that both grain size and L/D ratio following cold rolling and annealing are essentially insensitive to annealing temperature. Similar results were obtained for the combination of transverse rolling and annealing treatments.

As a corollary to this study, some of the as-received 0.275 inch thick sheet was annealed at 2000°F for 2 hours, then reduced 50% by cold rolling in the longitudinal direction, and then re-annealed at 2000°F (material state X). Specimens were then rolled 20 and 50% in the longitudinal and transverse directions (in separate rolling operations). A final annealing treatment of 2 hours at 2000°F was used. Figure A-2 indicates the effect of such material processing on the final grain size and L/D ratio. As shown for the previous material states of the TD-NiCr sheet, the processing-related variables of rolling and annealing had little effect on the grain structure in this material.

The effect of swaging deformation prior to annealing at 2000°F was also examined using the 0.275 inch thick TD-NiCr sheet (annealed at 2000°F for 1 hour) as the starting material. Swaging rods, 0.25 inch in diameter, obtained from this material, were oriented such that the rod axis was parallel to the original sheet rolling direction. Swaging deformations of 25 and 57% reduction in area were performed in steps of approximately 15% per pass. The grain size and L/D ratio for these materials following annealing is shown in Figure A-3. The average grain dimension decreased from the initial value of 6.3 microns with

increasing amounts of prior deformation to 2.4 microns at 57% reduction in area. The L/D ratio increased with prior deformation severity to a value of 3 at 25% reduction in area, then decreased slightly to a value of 2.7 at 57% reduction in area.

B_{1500}

It was indicated in the first section of this report that TD-NiCr 0.1 inch thick sheet could be transformed to a large grain structure by the annealing of the as-received material without further processing. An investigation of the effect of further thermo-mechanical processing on this large grain size material was conducted. A portion of this work constituted the Senior Project of C. Oldfather, an undergraduate in the Department.

The starting material for this examination was the as-received 0.1 inch thick TD-NiCr sheet after annealing at 1500°F for 90 hours (B_{1500}). The average grain size of this material was 0.23mm with an L/D ratio of 6. Elevated temperature tensile tests conducted on this material* revealed the following properties at 2000°F (1093°C): yield strength 14.4 ksi; tensile strength 14.6 ksi; ductility 1.8% elongation. Representative micrographs are shown in Figure A-4. The B_{1500} was cold rolled longitudinally with respect to the original rolling direction to reductions of 10, 28 and 50%. Rolling was performed in step reductions of approximately 5%. Following rolling, specimens of each reduction were annealed at 1500°F for 115 hours and also at 2000°F for 1 hour.

Figure A-5 shows the effect of rolling severity and annealing temperature on the grain size and L/D ratio following annealing. The specimens annealed at 1500°F were observed to have a steadily decreasing grain size with increasing

*Test conditions the same as those indicated on page 4 of previous section.

amounts of prior deformation. The L/D ratio increased slightly at moderate deformations to a value of 8.6 before decreasing to 2.2 at 50% reduction. The material annealed at 2000°F was shown to increase in both grain size and L/D ratio for moderate deformations (10-30%); this then decreased with increasing amounts of prior deformation. The maximum values of grain size and L/D ratio observed were those for 28% reduction preceding annealing at 2000°F. The average grain dimension and L/D ratio for this material state were 0.31mm and 18.6, respectively.

Since the combination of moderate rolling deformations (10-30% rolling reduction performed longitudinally with respect to the original rolling direction) and annealing at 2000°F for 1 hour produced an increase in the grain size and L/D ratio, it was felt that repeated combinations of the procedures described above would further increase the value of these properties. Three different processing methods were investigated; (1) the starting material, B_{1500}, was rolled 10% longitudinally, annealed (at 2000°F), re-rolled 10% in the same direction, (2) B_{1500} was rolled 10% longitudinally, annealed, rolled 10% in the transverse direction and annealed, and (3) B_{1500} was rolled 20% longitudinally, annealed, re-rolled 20% in the same direction and annealed. Table A-1 represents a summary of this investigation.

It was observed that repeated combinations of 10% longitudinal rolling reductions and annealing produced little change in the grain structure of the TD-NiCr sheet (B_{1500}). Alternate longitudinal and transverse rolling reductions of 10% followed by annealing treatments had the effect of increasing the grain size while decreasing the L/D ratio. However, the most effective method found to produce large, elongated grain structures in TD-NiCr sheet was the combination of 20% longitudinal rolling reductions separated by annealing treatments.

The method, as described, produced large grain structures of TD-NiCr (average grain dimension 0.50mm) with an L/D ratio of 30. Representative micrographs of this material state are shown in Figure A-6.

TABLE A-1

MULTIPLE PROCESSING METHODS USED ON

B_{1500} STARTING MATERIAL

Material Condition	Average Grain Dimension	L/D Ratio
B_{1500}	0.23mm	6.0
Schedule 1: B_{1500} rolled 10% long., annealed*, rolled 10% long., and annealed.	0.20mm	6.8
Schedule 2: B_{1500} rolled 10% long., annealed, rolled 10% trans., and annealed.	0.37mm	4.6
Schedule 3: B_{1500} rolled 20% long., annealed, rolled 20% long., and annealed.	0.50mm	30

*All annealing performed at 2000°F (1093°C) for 1 hour in air.

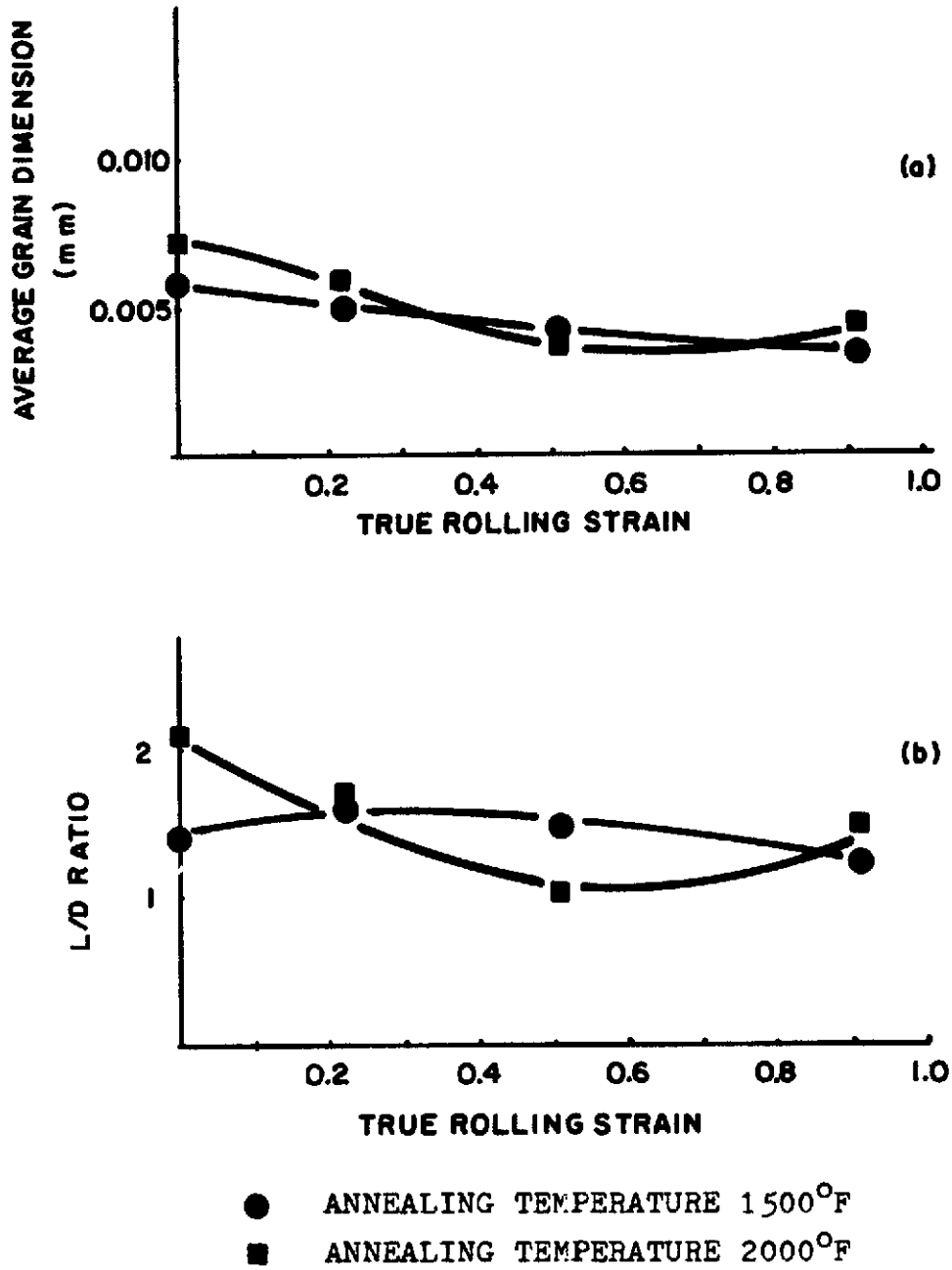


Figure A-1. The effect of deformation severity and annealing temperature on the average grain dimension and L/D ratio for 0.275" thick TD-NiCr sheet starting material.

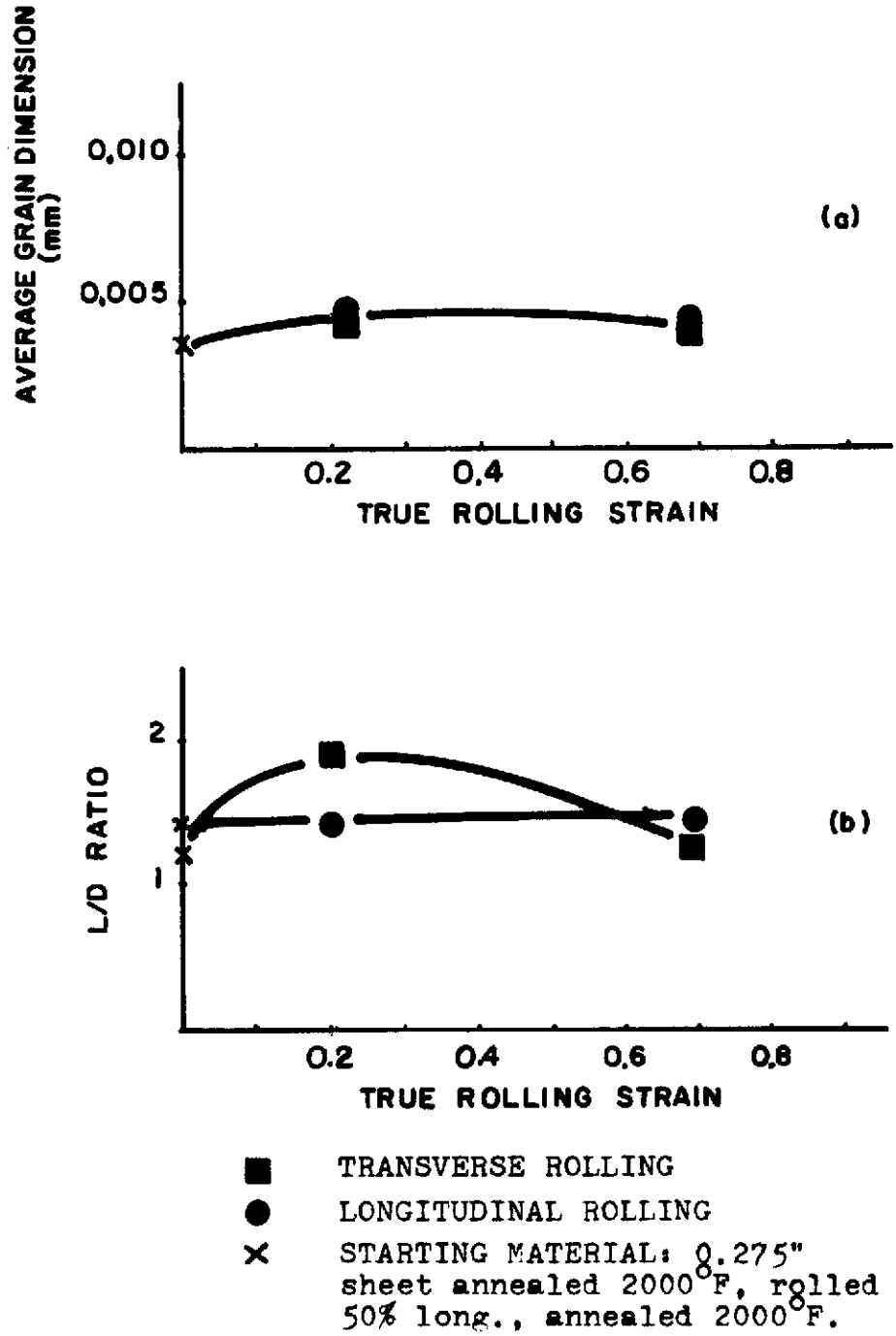


Figure A-2. Effect of deformation severity and mode on the average grain dimension and L/D ratio for material state X, starting material.

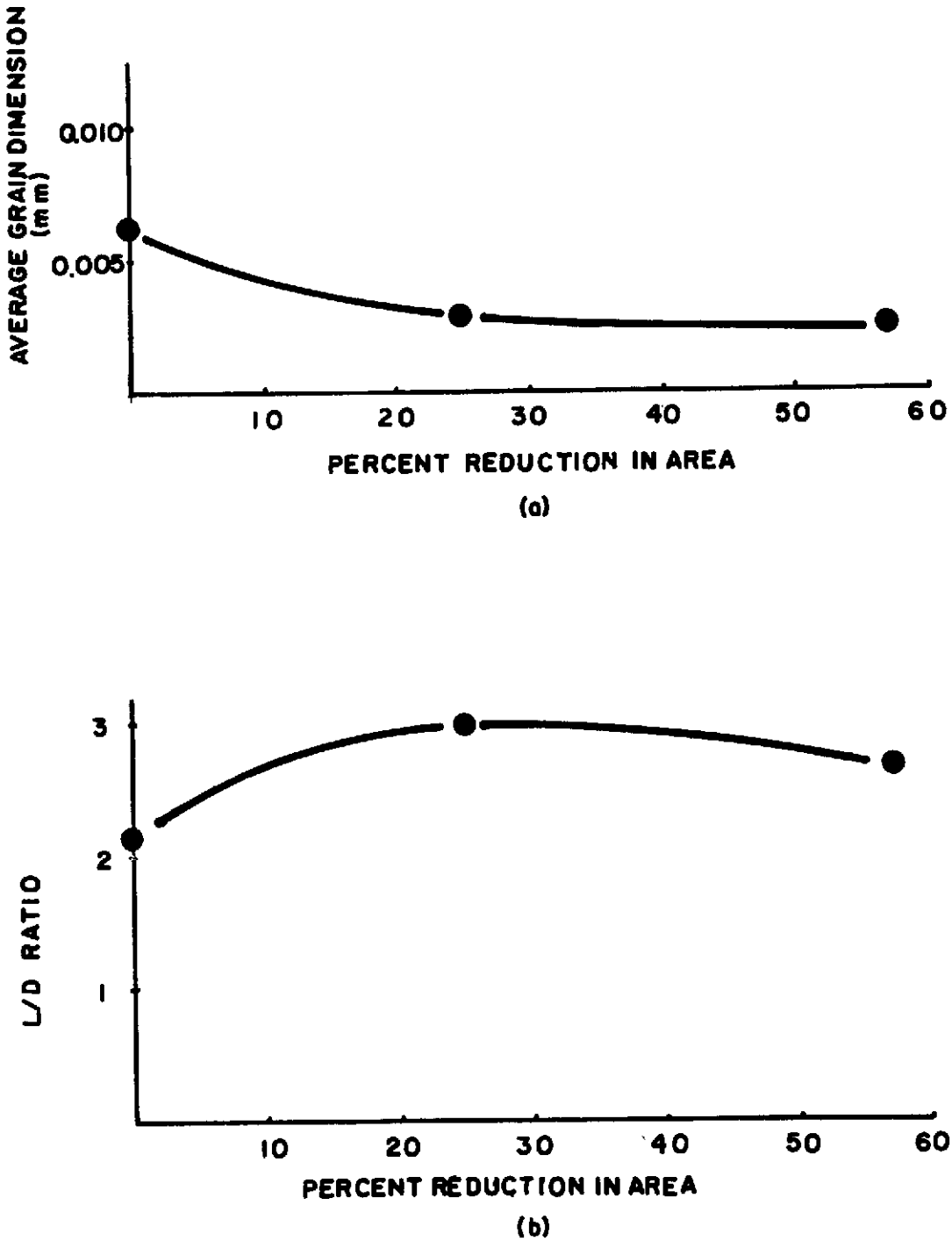
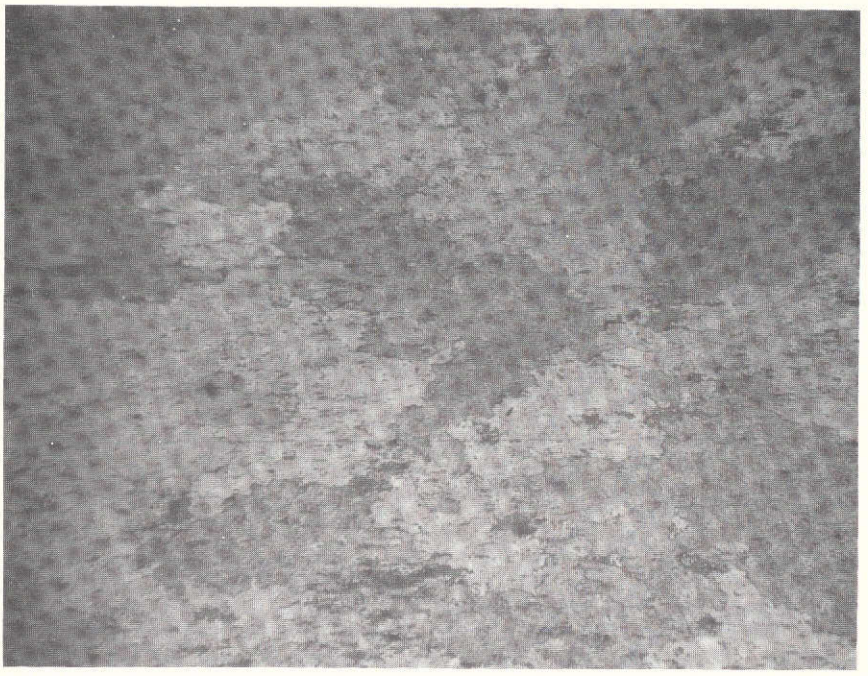
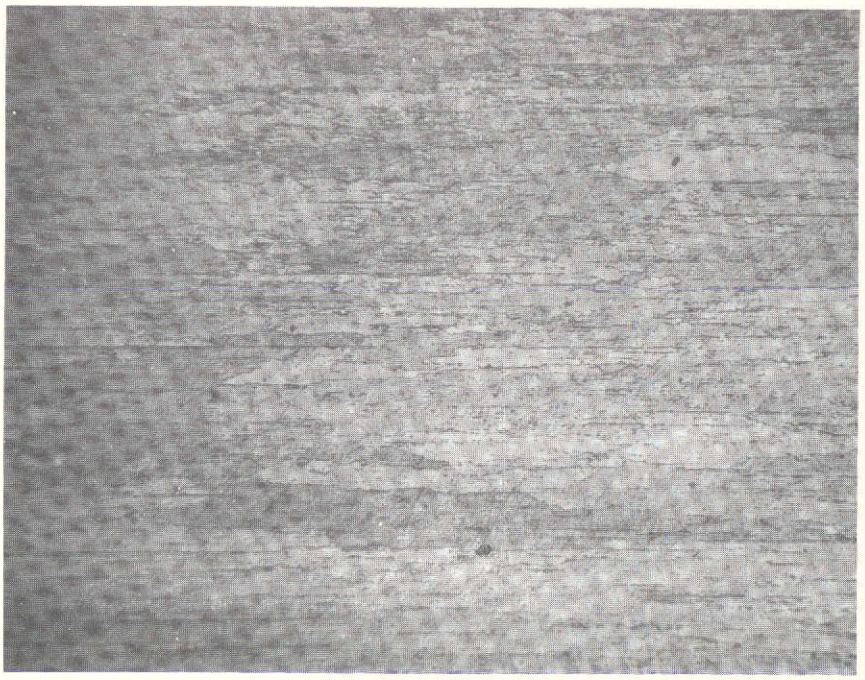


Figure A-3. Effect of swagging deformation and annealing at 2000°F on the average grain dimension and L/D ratio.



(a) (30X)



(b) (50X)

Figure A-4. Microstructure of the as-received material after annealing at 816°C (1500°F) for 90 hours. a) Rolling plane, b) Thickness plane, R.D. horizontal.

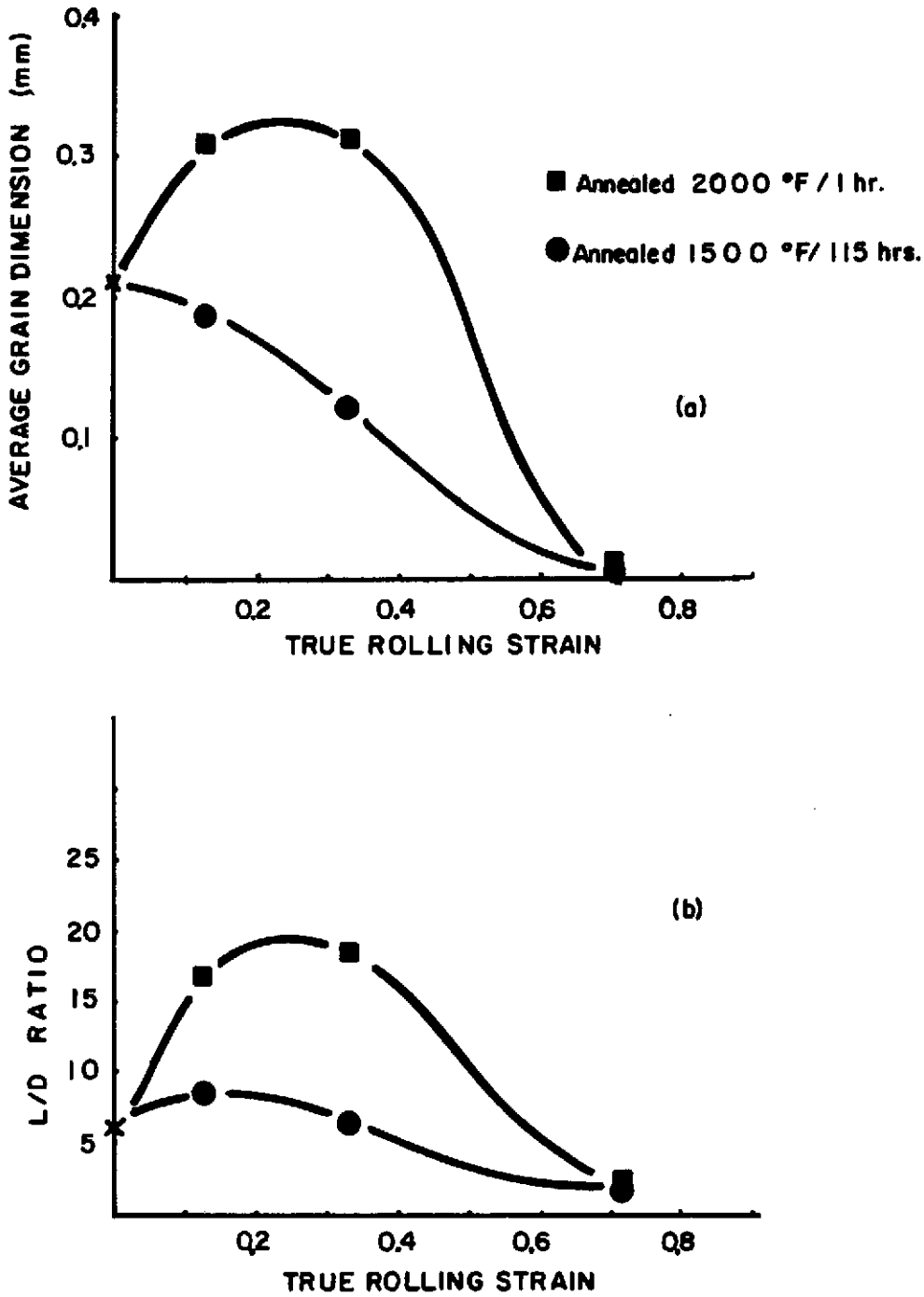
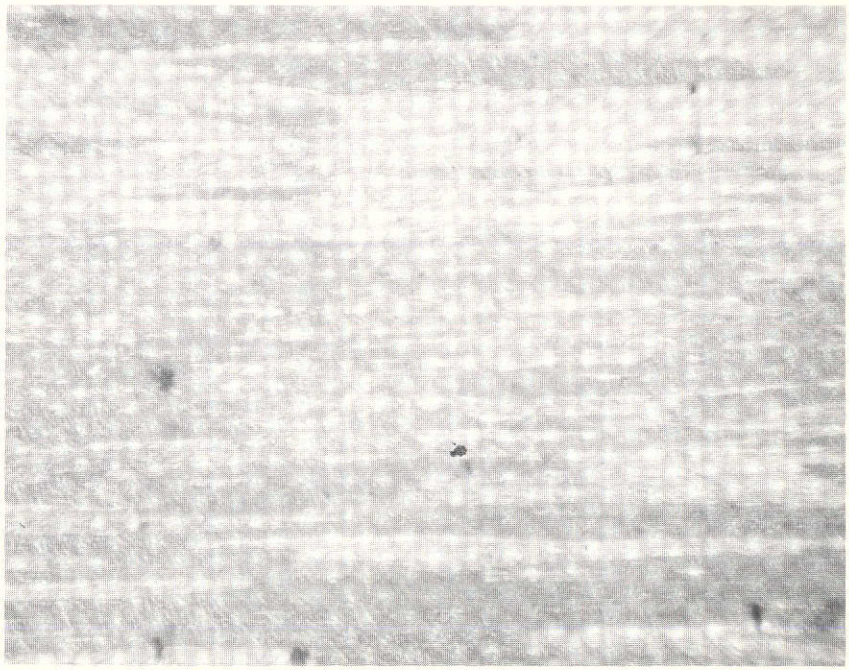


Figure A-5. The effect of rolling severity and annealing temperature on the average grain dimension and L/D ratio for B_{1500}, starting material.

(a) (10X)



(b) (50X)

Figure A-6. Microstructure of the as-received material after processing schedule 3. a) Rolling plane and b) Thickness plane, R.D. horizontal.

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